3rd Global Summit on Climate Changes and Sustainability

September 10-11, 2025 | Barcelona, Spain

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Comparative Study of Acid Mixtures for Sediment Digestion in TXRF: Focus on Heavy Metal Recovery and Accuracy

This study aims to optimize the sediment digestion method for chemical analysis by Total Reflection X-ray Fluorescence (TXRF) by testing several acid combinations used in the microwave-assisted digestion of a certified reference material (CRM) of sediment. Four digestion variants were investigated: aqua regia ($HNO_3:HCl$ = 2.5:7.5 v/v), concentrated HNO_3 (65%), an $HNO_3:H_2O_2$ mixture (7:3 v/v), and an $HNO_3:HCl:H_2O_2$ mixture (5:3:2 v/v/v). The analysis focused on the determination of Cr, Ni, Cu, Zn, and Pb. Each method's performance was evaluated by calculating the recovery rate, precision, repeatability, and accuracy.

Typically, for complete digestion of silicate matrices like Danube River sediments, hydrofluoric acid (HF) is recommended due to its ability to decompose the resistant mineral fraction. However, HF was deliberately excluded in this study due to the significant hazards associated with its handling and the potential for chemical interferences in TXRF analysis—such as the formation of volatile or insoluble compounds that can compromise measurement accuracy.

The results showed that no single digestion method was optimal for all analytes. Certain acid combinations were particularly effective for specific elements, while others yielded better performance for different targets. This variability underscores the importance of tailoring the digestion protocol to the specific elements of interest. The study thus provides a solid foundation for the rational selection of a digestion method suitable for Danube River sediments in the context of TXRF analysis.

Biography

Nina Nicoleta Lazar completed her PhD at the age of 30 years at Dunarea de Jos University of Galati, Romania. She is a research assistant at REXDAN Research Infrastructure. She has published 9 papers as first author and 18 papers as co-author in reputed journals, having an h-index of 10 on Google Scholar and Scopus, and 9 on WOS. She is part of the research team of three Horizon projects, with a research work experience of 7 years in multiple national and international research projects.